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(54) Title: IN-MOLD LABELS AND USES THEREOF

(57) Abstract -

This invention relates to an in-mold label comprising a core layer with a first and second surface and a heat seal layer on the first surface of the core layer, wherein the heat seal layer comprises a polyolefin, having a peak melt temperature of less than about 110 °C and where less than about 25 % of the polyolefin melts at a temperature of less than 50 °C as measured by differential scanning calorimetry. The label may also contain a skin layer on the second surface of the core layer. The invention also relates to plastic substrates bonded to the label. In another aspect, the present invention also relates to a process for in-mold labeling and a process for preparing an in-mold label. The labels and processes provide reduced amounts of one or more of the following: blisters, both before and after bonding, shrinkage, bagginess and gage bands.

Title:

IN-MOLD LABELS AND USES THEREOF

Technical Field of the Invention

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This invention relates to in-mold labels for plastic substrates. The invention provides labels which have reduced blistering. The invention also relates to plastic substrates containing the label and processes for applying the label and making the label.

Background of the Invention

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In-mold labeling has significant advantages over methods commonly used in the past to label plastic containers with polymeric labels. The most common of these previous methods involve the use of liner-carried pressure sensitive adhesive labels, or liner carried heat activatable adhesive labels. To produce the liner carried labels, a laminating step is performed to sandwich a layer of adhesive between a web of label stock and a web of silicone-coated paper which is to function as a carrier or release liner, the label stock is printed, the ink is dried by heating elements or ultraviolet radiation, separate labels are cut from the label stock by passing the combination through a rotary-die or flat-bed cutting station, and the matrix of waste or trim label stock surrounding the labels is stripped and discarded or recycled. Use of these types of methods results in high costs due to the use of a release liner, and the ecological difficulties in disposing of the liner and the trim.

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In contrast, in-mold labeling avoids the use of any release liner or carrier. During in-mold labeling with polymeric labels, self-supported or free-film polymeric label stock is combined with heat-activatable adhesive, printed, die-cut and then arranged for deployment, as by being magazine-loaded as a series or stack of linerless labels, or by other means. The polymeric labels are then sequentially deployed on the molding surface of a blow mold to be bonded onto successive hot plastic substrates or containers. The blow molded parisons are expanded against the molding surface and the in-mold

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conditions, if the in-mold adhesive is applied prior to orientation as in coextruded film processes.

Conventional polymers have very broad molecular weight distributions,

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which means there will be fractions that have much lower melting points than the peak melting point. These fractions, called the low molecular weight tails, are significantly reduced in metallocene catalyzed polymers or in polymers which are distilled or fractionated to remove the low molecular weight tail. Hence polymers that have more ideal peak melting temperatures can be used, enhancing adhesive performance.

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The low molecular weight tail mentioned above causes "plate-out" problems which require frequent down-times for cleaning up the machine. In "plate-out", the low molecular weight tail separates from the main fraction of the film under the high temperature, pressure and shear in processing and condenses on the metal surface of rollers, dies and metal rolls. This condensed material attracts dirt, dust and suspended additives in the formulation and causes surface contamination. Thus, as the plate-out builds up on the machine, the machine needs to be stopped and cleaned periodically. Polymers without the low molecular weight tail, such as those prepared using metallocene catalysts, greatly decrease this downtime, from about once every 3 hours to about once every 24 hours, improving economics, throughput, productivity and reducing waste from shutting down the processing line. The reduced plate out allows an oriented film to be annealed at a higher temperature, which reduces film shrinkage.

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Similar to the low molecular weight tail, there is also a high molecular weight tail present in polymers. This tail has a melting point which is substantially higher than the peak fraction. In the in-mold labeling process, as the hot plastic substrate or container comes into contact with the adhesive, the high molecular weight tail requires higher temperatures to activate. This higher temperature results in (1) very high cycle times since high temperatures also need long cooling times, which decreases productivity, or (2) non-uniform activation of adhesive, which causes blisters. In practice, both problems are

with a metallocene catalyst. The label may also contain a printable skin layer on the second surface of the core layer. The invention also relates to plastic substrates bonded to the label.

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In another aspect, the present invention also relates to a process for inmold labeling comprising the steps of: forming a label comprising a core layer
with a first and second surface and a heat seal layer on the first surface of the
core layer, wherein the heat seal layer comprises a polyolefin, having a peak
melt temperature of less than about 110°C and wherein less than about 25%
of polyolefin melts at a temperature of less than 50°C as measured by
differential scanning calorimetry; inserting the label into a mold for producing a
plastic substrate with an inside and outside surface; and forming a plastic
substrate in the mold with sufficient heat to bond the heat seal layer on the label
to the outside surface of the plastic substrate. In another aspect, the invention
relates to a process for preparing an in-mold label comprising the steps of
coextruding a multi layer film comprising a core layer having a first and second
surface and a heat seal layer on the first surface of the core layer and annealing
the film at a temperature above 75°C. In another aspect, the film is stretched
in one or two directions.

The labels and processes provide reduced amounts of one or more of the following: blisters, both before and after bonding, shrinkage, bagginess and gage bands.

Brief Description of Drawings

Fig. 1 is a cross sectional view of a label.

Fig. 2 is a representation of an embodiment of the method of preparing labels.

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Description of the Preferred Embodiments

As used in the claims and specification, peak melt temperatures and percentage of melting material is determined by differential scanning calorimetry. The test is performed on a TA Instrument model 912 Differential Scanning Calorimeter. The heating cycle is from -60°C to 300°C with a heating rate of 10°C/minute. The heat absorbed is measured and plotted as is known to those

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about 35% to about 85% by weight. In one embodiment, the polyolefin is present in an amount from about 40% up to about 99%, or from about 50% to about 95%, or from about 60% up to about 90% by weight. In another embodiment, the polyolefin is present in an amount from about 20% up to about 70% or from about 25% to about 60%, or from about 30% up to about 50% by weight. Here as well as elsewhere in the specification and claims, the range and ratio limits may be combined.

The polyolefin may be a homopolymer or a copolymer. The olefins which may be used to prepare the polyolefins include those having from about two to about ten, or from two to about eight, or from about two to about four carbon Examples of useful olefins include ethylene, propylene, butylene, methyl-pentene, hexene, octene, etc. In a preferred embodiment, the polyolefin is a homopolymer or copolymer derived of ethylene, propylene or butylene. In one embodiment, the polyolefin is an ethylene homopolymer or an ethylene copolymer. The copolymer is prepared from ethylene, propylene, or butylene and an olefin having from about 3 to about 100 or from about 4 to about 30 carbon atoms. In one embodiment, the olefin has from about three to about twelve, or from about four to about ten carbon atoms. In another embodiment, the olefin has from about 10 to about 100, or from about 12 to about 30 carbon atoms. In one embodiment, the olefin used to prepare the copolymer is an alpha-olefin. Examples of useful olefins include propylene, butylene, pentene, 4-methyl-1-pentene, hexene, heptene, octene, nonene, decene, undecene, dodecene, tetradecene, hexadecene, octadecene, and docosene. Typically, the olefin is present in an amount from about 1% up to about 50%, or from about 5% to about 30%, or from about 7% up to about 25% by mole. Examples of copolymers of ethylene include ethylene/propylene copolymers, ethylene/butylene copolymers, ethylene/hexene copolymers, ethylene/octene copolymers and ethylene/dodecene copolymers of ethylene and olefins, such as alpha-olefins are disclosed in U.S. Patents 5,475,075, issued to Brant et al, and 5,530,054, issued to Tse et al. These patents are incorporated by reference for their disclosure of ethylene olefins and processes for making the same.

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terephthalate (PBT), polyethylene terephthalate (PET), thermoplastic polyesters and mixtures of two or more thereof. The blend preferably contains from about 1% to 80%, or preferably from about 5% to about 75%, or from about 10% to about 60% by weight of the additional polymers. Such a blended adhesive layer would have the advantages of combining the properties of the metallocene polyolefin with the throughput and processability of the other component. The other components may also be chosen such that the adhesive is more compatible with the substrate to which it is bonded, for example, polypropylene or polyethylene terephthalate containers.

The heat seal layer may contain an antiblock or an antistatic additive. The antiblock additives include natural silica, diatomaceous earth, synthetic silica, glass spheres, ceramic particles, etc. The antiblock additive may be added as a concentrate. An example of an antiblock concentrate is manufactured by A. Schulman Inc. of Akron, OH under the product name Polybatch F-20. The Polybatch F-20 is an antiblock concentrate containing 20 weight percent natural silica based in low density polyethylene, and is designed for use in polyethylene Both the particle size and the dispersion of this antiblock concentrate has been optimized to offer effective coefficient of friction values when used in conjunction with standard slip concentrates. The Polybatch F-20 material has the following material properties (which are based on its technical data sheet): melt index of the carrier resin of 9+/- 4 grams/10 minutes; melt index of concentrate of 7 +/- 3 grins/10 minutes; ash (percent natural silica) of 20+/- 2 percent; moisture retention (Karl fisher @ 190 °C) of 1000 ppm maximum; and pellets per gram of 46+/- 5. When the antiblock is present the antiblock is present in an amount from about 10,000 to about 60,000, or from about 20,000 to about 50,000, or from about 40,000 ppm.

The antistatic additives include an amine or an amide or a derivative of a fatty acid. The antistatic is incorporated in the adhesive-containing or base layer charge and uniformly blended therewith. The amount of antistatic used may be varied for particular formulations and processing conditions. Typically from about 0.5% to about 15%, or from about 2% to about 10%, or about 5% by

polystyrene, polypropylene, cyclic olefins, polyethylene methyl acrylate, acrylonitrile, butadiene styrene polymer, polyethylene vinyl alcohol, polyethylene vinyl acetate, nylon, polybutylene, polyurethane, polysulfone, polyvinylidene chloride, polycarbonate, polymethyl pentene, styrene maleic anhydride polymer, styrene acrylonitrile polymer, ionomers based on sodium or zinc salts of ethylene/methacrylic acid, polymethyl methacrylates, cellulosics, fluoroplastics, polyacrylonitriles, polybutylene terephthalate (PBT), polyethylene terephthalate (PET), thermoplastic polyesters and mixtures of two or more thereof.

In one embodiment, the core layer is composed of a polyolefin, such as low, medium, or high density: polyethylene, polypropylene or polybutylene or copolymers of ethylene, propylene or butylene with an olefin, such as those discussed above. The polyolefin may be prepared using a metallocene catalyst. The polyolefin may be prepared from a copolymer of propylene and an alpha olefin, such as those containing from about two to about ten carbon atoms, and including ethylene, butylene, hexene and octene. An example of a useful polypropylene copolymer is sold by Union Carbide Corporation of Houston, Texas, under the product number 5A97. The 5A97 product has the following characteristics: a melt flow index of 3.9 grams/10 minutes; a melting point of 162 °C; tensile strength at yield of 5350 psi; elongation at tensile yield of 8%; and a flexural modulus (1/a secant) of 230,000 psi. The propylene copolymers include random propylene copolymers which have from 3 to about 5% ethylene.

An example of propylene copolymer includes a random copolymer polypropylene which is sold by Union Carbide Corporation of Houston, Texas, under the product name 6D20. The 6D20 random copolymer polypropylene has the following characteristics: a melt flow of 1.6-2.2 grams/10 minutes; a density of 0.890 grams/cc; a tensile yield strength of 3600 psi; a yield elongation of 14%; a flexural modulus (tangent) of 130,000 psi; a vicat softening temperature of 134°C; and a melting temperature of 147 °C. Another random copolymer polypropylene is 5E98 available from Union Carbide Corporation.

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material usage. Reduction of thermal conductivity is rendered irrelevant due to the superior adhesion obtained with metallocene catalyzed polymers.

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In another embodiment, the core layer may include a calcium carbonate concentrate. The calcium carbonate concentrate is preferably used with a polyolefin homopolymer, such as a polypropylene homopolymer. The calcium carbonate concentrate is present in an amount from about 30% to about 85%, or from about 40% to about 80%, or from about 50% to about 70% by weight of the core layer. An example of a calcium carbonate concentrate is composed of polypropylene with 40 weight percent calcium carbonate mineral filler, the material has a melt index of 3.0 minimum to 6.0 maximum (ASTM D1238); ash of 40.0+/- 2.0%, volatiles of 500 ppm max.; and a bulk density of 730+/- 50 g/1. The calcium carbonate concentrate is sold by A. Schulman Inc. of Akron, Ohio under the product name Polybatch PF92D. The calcium carbonate concentrates are used to prepare voids in the core layer as is discussed below.

film materials, or by the orientation of an incompatible two phase system. More preferably, the film is produced by the orientation of an incompatible two phase system. Oriented incompatible two phase systems can either be uniaxially oriented or biaxially oriented. Most preferably, the film is uniaxially oriented.

While each of the layers of the film including the skins may contain voids,

Voided films can be produced by the use of blowing agents in the polymer

preferably, the skin layer and/or the heat seal layers contain essentially no voids. Essentially no voids preferably means that the skin layers contain less than about 10% by volume of voids, and more preferably less than about 3% by volume of voids. Preferably, one of the layers of the core contains voids, and more preferably the entire core contains voids.

In another embodiment, the core is not voided and is composed of polymers or blends of polymers, such that the core is optically transparent. Material for such core includes the polyolefins (i) and addition polymers (ii) described above.

The film can be manufactured by those processes known to those in the art such as casting, coating or extrusion. U.S. Patents 5,186,782, issued to

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the stiffness in the unoriented direction (cross direction for extruded films) is less than about 0.75 times the stiffness in the oriented direction. Still preferably the stiffness in the unoriented direction is less than about 0.6 times the stiffness in the oriented direction, more preferably less than about 0.5 times.

For an incompatible two phase system, the void initiating particle or phase material, as indicated above, should be, at least partially, incompatible with the core material, at least at the conditions of uniaxial orientation. Typical void initiating particles materials can be organic or inorganic. Examples of organic void initiating particles include but are not limited to polyamides; nylons; high density polyethylene; polyesters such as polyethylene terephthalate; acetals; polypropylene homopolymer; and acrylic resigs. Examples of inorganic void initiating particles include but are not limited to solid and hollow preformed glass spheres, metal beads or spheres; ceramic particles; titanium dioxide; calcium carbonate; barium sulfate; chalk; silicon dioxide; and clay. The preferable inorganic void initiating particle materials are mica, clay and chalk; more preferable material is ceramic spheres; and the most preferable material is calcium carbonate. The preferred mean particle size of the void initiating particle or phase is from about 0.1 to about 12μ . The more preferred mean particle size of the void initiating particle or phase is from about 0.5μ to about 5μ . The most preferred mean particle size of the void initiating particle or phase is from about 1 to about 3μ .

As indicated above, certain low cost filler materials such as calcium carbonate can be used as void initiating particles. Therefore, the greater the amount of void initiating particle, the lower the cost of materials in the film. Preferably, the void initiating particles or phase is present in an amount greater than about 20 weight percent of the voided layer prior to orientation. More preferably, the void initiating particles or phase is present in an amount greater than about 25 weight percent of the voided layer prior to orientation. Most preferably, the void initiating particles or phase is present in an amount greater than about 30 weight percent of the voided layer prior to orientation.

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extrusion through the multifeed coextrusion die 18 as illustrated in Figure 2.

In this embodiment, the hot stretching is performed at a temperature equal to or above the softening temperature of the film and provides film orientation. During hot stretching the extrusion or coextrusion die is preferably maintained between from about 200°C to about 230°C, and more preferably at about 220°C. The extruded film is cast onto a casting roll 21 which is preferably maintained between from about 20°C to about 50°C, and more preferably at about 30°C, and is provided with an air knife 19. The film continues around the casting roll 21 and then passes to the chill roll 22 which is preferably maintained from about 20°C to about 70°C, and more preferably at about 66°C. The film continues around the chill roll, trains through the rolls 24, and enters the uniaxial (or machine) direction orientation unit (MDO) 25. The film is preferably moved at a rate of between from about 20 to about 35 feet per minute past all these rolls, and more preferably at about 30 feet per minute.

Within the MDO unit, the film is stretched and stiffened in the machine direction. The film is passed around a first pre-heat roll 26 and then around a second pre-heat roll 28. These rolls are maintained at between from about 90°C to about 120°C, or about 110°C, and from about 60°C to about 100°C, or about 80°C, respectively, and at this point the film continues to move at between from about 20 to about 35 feet per minute, and more preferably at about 30 feet per minute. After leaving the second pre-heat roll the film tracks on the slow draw roll 31, maintained at a temperature from about 80°C to about 120°C, or about 100°C, still preferably moving at between from about 20 to about 35 feet per minute, and more preferably moving at about 30 feet per minute. The film is then pulled to the fast draw roll 32, maintained at about 75°C to about 105°C, or about 85°C, which preferably advances the stock at the rate of between from about 120 to about 210 feet per minute, and more preferably at about 180 feet per minute. Therefore in this particular preferred embodiment described, the film is

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inside the mold cavity during the molding process for producing a plastic container with the adhesive side facing the outside surface of the hot plastic container. As the plastic is molded, the heat activates or melts the adhesive on the label, and the label and the plastic container form a permanent bond.

Examples

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In order that persons in the art may better understand the practice of the present invention, the following Examples are provided by way of illustration, and not by way of limitation. Additional background information known in the art may be found in the references and patents cited herein, which are hereby incorporated by reference.

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These examples are samples of film typically used as in-mold labels. For in-mold labeling this film would typically be converted into labels for application onto plastic containers during the process of forming these containers. Table 1 contains examples of formulations useful as heat seal layers. Table 2 contains examples of formulations useful as core layers. Table 3 contains examples of formulations useful as skin layers. Table 4 contains examples of two or three layer films where all the layers are produced by a coextrusion process.

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Table I shows the composition of the heat seal layer by weight percent of components. The ethylene/octene copolymer is AFFINITY PF 1140, which has 14.0% octene, a melt index of 1.6 dg/min. and a density of 0.895 gm/cc. HPP is a homopolymer polypropylene sold by Union Carbide Corporation of Houston, Texas, under the product number 5A97. The 5A97 product has the following characteristics: a melt flow index of 3.9 grams/10 minutes; a melting point of 162 °C; tensile strength at yield of 5350 psi; elongation at tensile yield of 8%; and a flexural modulus (1/a secant) of 230,000 psi. The RnCoPP is a random copolymer polypropylene sold by Union Carbide Corporation of Houston, Texas, under the product name 6D20. The 6D20 random copolymer polypropylene has the following characteristics: a melt flow of 1.6-2.2 grams/10 minutes; a density of 0.890 grams/cc; a tensile yield strength of 3600 psi; a yield elongation of 14%; a flexural modulus (tangent) of 130,000 psi; a vicat softening temperature of 134°C; and a melting temperature of 147°C. The

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data sheet): melt index of the concentrate of 11-18 grams/10 minutes; and moisture retention (Karl Fisher 0 190 °C) of 1000 ppm minimum.

The materials for the heat seal layer are melted and mixed in a 2 1/2 inch extruder manufactured by Davis Standard of Pawcatuck, CT with a screw of a L/D ratio of 24: I. The extruder contains six temperature zones which were maintained at 177, 205, 216, 222, 222, and 224°C, respectively.

The print skin layers of the three layer film are melted and mixed in a 2 1/2 inch extruder equipped with a screw of a L/D ratio of 24:1. This extruder is manufactured by Davis Standard of Pawcatuck, CT. The extruder contains six temperature zones which where are maintained during melting and mixing at 177, 205, 222, 222, 227, and 227°C, respectively.

The cores of the films are melted and mixed in a 130mm extruder equipped with a screw of a L/D ratio of 34:1. This extruder was manufactured by Davis Standard of Pawcatuck, CT. The extruder contains eight temperature zones which are maintained during melting and mixing at 196, 199, 202, 207, 210, 213, 216, and 227°C, respectively. The calcium carbonate of the core material is dried prior to mixing in an oven manufactured by Conair Franklin of Franklin, PA at 80 °C for four hours to insure that the concentrate material contains little or no moisture.

The three extruders feed a multilayer feedblock which then feeds a single manifold coathanger die. Both the feedblock, die and adapter pieces which connects the extruders, feedblock and die together are held at a constant temperature of 218°C. The three layer films are extruded onto a casting roll which is maintained at 30 °C, and are provided with an air knife for further cooling the film and followed by a second cooling roll at 66°C. The film is then stretched uniaxially in the machine direction by being run through an orientation unit which orients the film in the machine direction (MD). The orientation unit consists of a number of rolls the first two of which are used for pre-heating the film prior to stretching. These rolls are maintained at 110 °C and 80°C, respectively so that a substantial portion of the thickness of the film is heated. The film is stretched between the slow draw roll, maintained at 100°C (moving

Table 2 (Core Layer)

| | | | | ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~ | | | |
|---------|--------|-----------|-----|--|-------------|-------------|--|
| Example | RncoPP | Ethylene/ | EVA | HPP | Titanium | Calcium | |
| | - | octene | | , | Concentrate | Carbonate | |
| • | | copolymer | | | | Concentrate | |
| | | | | | | | |
| 1 | 100 | | | | | | |
| 2 | 80 | 5 | *** | | 15 | | |
| .3 | 85 | | 5 | | 10 | | |
| 4 | 75 | | 10 | | 15 | | |
| 5 | 70 | | 15 | | 15 | | |
| 6 | 55 | 25 | 10 | | 10 | | |
| 7 | | | | 10 | | 90 | |
| 8 | | | | 40 | | 60 | |
| 9 | | 10 | | 40 | | 50 | |
| ··· 10 | | | | 20 | | 80 | |
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Table 4

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|------------|---|--------------|---|---|---|---|---------------------------------------|-----|
| | Α | В | С | D | Е | F | G | Н |
| Skin Layer | | : | | | | | | |
| (Table 1) | | | | | | | | |
| Ex. 1 | X | | | | | | | |
| Ex. 3 | | Х | | | | X | | Х |
| Ex. 4 | | | Х | X | | | | |
| Ex. 7 | | | | | Х | | Х | |
| | | | | | | | | |
| Core | | - | | | | - | V | |
| Layer | | | | | | | | |
| (Table 2) | | | | | | | | |
| Ex. 1 | X | | | | | Х | Х | |
| Ex. 5 | | Х | | X | | | | ~~~ |
| Ex. 10 | | | Х | | Х | | | X |
| | | | | | | | | |
| Skin Layer | | | | | | | | |
| (Table 3) | | | | | | | | , |
| Ex. 1 | | Х | | | | | X | |
| Ex. 3 | | | Х | | Х | × | | Х |

While the invention has been explained in relation to its preferred embodiments, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to be understood that the invention disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

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- 9. The label of claim 1 wherein the heat seal layer is a blend of the polyolefin and ethylene vinyl acetate.
- 10. The label of claim 1 wherein the heat seal layer is substantially free of ethylene vinyl acetate.
- 11. The label of claim 1 wherein the polyolefin is a low density polyethylene, polypropylene or polybutylene.
- 12. The label of claim 1 wherein the heat seal layer is free of carrier resins.
- 13. The label of claim 1 wherein the label has a shrinkage of less than about 5%.
- 14. The label of claim 1 wherein the label has a thickness of less than about 7 mils.
- 15. The label of claim 1 further comprising a printable skin layer on the second surface of the core layer.
 - 16. The label of claim 1 wherein the label is optically opaque.
 - 17. The label of claim 1 wherein the label is optically transparent.
- 18. An in-mold label comprising a core layer with a first and second surface, a heat seal layer on the first surface of the core layer, and a printable skin layer on the second surface of the core layer, wherein the heat seal layer comprises a polyethylene or an ethylene copolymer having a peak melt temperature of less than about 110°C and wherein less than about 25% of polyolefin melts at a temperature of less than 50°C as measured by differential scanning calorimetry.
- 19. The label of claim 18 wherein the polyolefin is an ethylene copolymer derived from ethylene and an olefin having from about 3 to about 12 carbon atoms.
- 20. The label of claim 18 wherein the polyolefin is an ethylene copolymer derived from ethylene and one selected from the group consisting of propylene, butylene, pentene, 4-methyl-1-pentene, hexene, heptene, and octene.
- 21. The label of claim 18 wherein the polyolefin is prepared with a metallocene catalyst.

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29. A process for preparing an in-mold label comprising the steps of coextruding a multi layer film comprising a core layer having a first and second and a heat seal layer of the first surface of the core layer and wherein the heat seal layer comprises a polyolefin having a peak melt temperature of less than about 110°C and where less than about 25% of the polyolefin melts at a temperature of less than 50°C as measured by differential scanning calorimetry, stretching the multilayer film is one or two directions, and annealing the film at a temperature above 75°C.

